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IS 12114-2 (1987): Gold Alloy Coverings on Watch Cases and their Accessories, Part 2: Determination of Fineness, Thickness and Corrosion Resistance [PGD 23: Horology]

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(Reafirmed - 2012)

Indian Standard

SPECIFICATION FOR GOLD ALLOY COVERINGS ON WATCH CASES AND THEIR ACCESSORIES

PART 2 DETERMINATION OF FINENESS, THICKNESS AND CORROSION RESISTANCE

1. Scope — Prescribes methods to determine fineness, thickness and corrosion resistance for gold alloy coverings on watch cases, their accessories and crown including bracelets when they are permanently attached to the case.

2. Definitions — For the purpose of this standard, the definitions given in IS : 3554-1966 'Glossary of terms relating to electroplating' and the following definitions shall apply.

2.1 *Significant Surface* — That area of the article covered or to be covered by the coating and for which the coating is essential for the appearance or serviceability of the components. In case of no agreement has been made between the supplier and the customer, 'significant surface' will be any surface which can be touched by a 5-mm diameter ball.

3. Determination of the Fineness

3.1 Fineness shall be measured on a gold alloy covering which is separated from the base metal. The gold alloy covering shall be separated by the method specified in Appendix A. The method used to separate the gold covering from the base metal shall in no way affect the fineness of the gold covering.

3.2 *Method*

3.2.1 *Basic method* — The basic method shall be by chemical analysis by reduction in aqueous solution, such as sulphur dioxide or any other suitable reducing agent.

3.2.2 *Secondary methods* — The fineness may also be determined by any one of the methods specified in **4.2** of IS : 3266-1982 'Specification for electroplated coatings of gold for general engineering purposes (*first revision*)'. Other methods such as:

a) X-ray fluorescence analysis;

b) Touch stone (to be used to determine minimum fineness); and

c) Other physico-chemical methods.

Any method used shall be capable of measuring fineness with in an accuracy of 50 parts per thousand.

4. Determination of Thickness

4.1 The following methods are recommended:

a) Dissolution method and chemical analysis for any thickness of gold alloy covering (average thickness); and

b) Microsection method for a thickness of 5 μm (−20 percent) and above (local thickness).

The microsection method specified in **3** of IS : 3203-1982 'Methods for testing local thickness of electroplated coating' shall be used as referee method.

4.1.1 Where there is no dispute, the following secondary methods may be used:

a) Dissolution and measurement by the micrometer method;

b) Beta-ray backscatter;

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c) X-ray fluorescence;
d) Touchstone (preliminary method only);
e) Interferometric method.

For guidance, refer to Appendix B.

5. Porosity and Corrosion Resistance

5.1 Unless otherwise agreed to, plating/covering shall be tested by one of the corrosion tests given in Appendix C depending upon the basis metal and under coating.

APPENDIX A

(*Clause* 3.1)

REMOVAL OF GOLD COATING FROM BASIS METAL AND UNDER COATS (IF ANY)

A-1. Introductory Notes

A-1.1 In order to bring the gold alloy covering to a form suitable for analysis and in order to determine its mass, it is necessary to separate it from the basis metal. The gold content is then determined by one of the methods described in **3.2.1** and **3.2.2.** Since complete mechanical separation is possible only in the minority of cases, this process is carried out by dissolution of the basis metal. In order to avoid any attach on the gold alloy covering, the time of exposure to the acid should be kept minimum.

A-1.2 The corners and edges of the test sample or part of it shall be chamfered by filing, etc, in order to increase the area of attack.

A-1.3 The basis metal is first removed, as far as possible, by mechanical means (by filing, milling, turning, etc) and the residual base metal dissolved in acid.

A-1.4 A sufficient quantity of the gold alloy covering is removed, as far as possible by mechanical means (for example, by scraping) and any adhering basis metal removed by dissolution in acid.

A-2. Dissolution of the Basis Metal

A-2.1 For copper nickel and iron alloy, which are not passivated, nitric acid, ρ 1·1 g/ml (one volume of concentrated nitric acid to 4 volumes of distilled water) is used. For gold alloy coverings of inferior fineness to 700 thousandths, a more dilute nitric acid, for example δ 1·05 g/ml (one volume of concentrated nitric acid to 9 volumes of distilled water) should be used. The time for complete dissolution will be longer than in the more dilute acid. If a problem appears (due to presence of tin) concerning the solution of the basis metal, 2 percent of flurohydric acid or 5 percent of fluroboric acid can be added to the solution to avoid this problem.

A-2.2 For stainless steel, hydrochloric acid, ρ 1·125 g/ml (5 volumes of concentrated hydrochloric acid to 3 volumes of distilled water is used, aluminium alloys may be dissolved in 10 percent (*m*/*m*) sodium hydroxide solution. Generally, such samples have undercoats of copper and/or nickel which are not attacked by sodium hydroxide. These undercoats must, therefore, be dissolved in dilute nitric acid as described above after complete dissolution of the aluminium basis metal.

In each case, the basis metal and undercoats (if any) are dissolved by heating the dissolving medium to temperature of 90 to 95°C. After complete dissolution of the basis metal, the solution is decanted, the residual coating washed several times first with dilute nitric acid and then with distilled water, and dried at 110°C.

A-2.3 Certain gold alloy covering will disintegrate completely after dissolution of the basis metal. In this case the solution is filtered through a weighed filtering crucible, the residual gold alloy coating is washed and dried as described in **A-2.2.**

A-2.4 Special care should be taken with tin-containing basis metals as the stannic hydroxide formed cling sobstinately to the gold alloy covering. In order to avoid any excessive precipitation of stannic hydroxide the nitric acid should be renewed several times. Boiling of the solution must be avoided. After complete dissolution of the basis metal, the remaining gold alloy covering is washed with distilled water and then treated with hot hydrochloric acid (1 volume of concentrated hydrochloric acid to 9·volumes of distilled water) to ensure complete dissolution of the adhering stannic hydroxide. The remaining gold alloy covering is washed and dried as described in **A-2.2**.

APPENDIX B

(*Clause* 4.1.1)

DETERMINATION OF COATINGS THICKNESS

B-1. Introduction Notes

B-1.1 The methods given in this Appendix are those which are considered to have an adequate accuracy when properly used with test specimens suitable for the particular methods. If a referee method is required it shall be as given in **B-3** or the method chosen shall be one which is expected to yield the most reliable results. Considering such factors as coating thickness, shape and size of component basis material, etc. Other test methods may be used, if it may be demonstrated that they have an accuracy of ±10 percent or better, or that they are as good as or better than the methods given in this Appendix for the particular application.

B-2. Dissolution Method and Chemical Analysis

B-2.1 *Principle* — Chemical or electrochemical dissolution of the substrate without attack of the gold coating. Calculation of the average thickness of the coating over a small area from the area, mass and density of the coating.

B-2.2 *Test Specimen* — Carefully punch or cut the test specimen from the article. The shape and size of the specimen shall be such that the area of gold may be measured with an accuracy of 2 percent or better. The mass of gold may be subsequently determined with an accuracy of 2 percent or better. It may be necessary to square off and sand the edges to remove gold smeared over the edge by the cutting operation.

B-2.3 *Procedure*

B-2.3.1 Measure the area of coating with an accuraccy of 2 percent or better.

B-2.3.2 Remove as much as possible of the basis material by mechanical means before stripping in order to minimize potential attack on the gold coating.

B-2.3.3 Dissolve the substrate material without attacking the coating. For many substrates, nitric acid ($\delta = 1.42$), diluted with three volumes of water may be used at approximately 25°C but this acid may dissolve some of the alloying elements in the case of some alloy coatings.

B-2.3.4 Remove the gold, dry at 110°C for approximately 30 minutes, and weigh to an accuracy of 2 percent or better. If the gold breaks up into small pieces, it may be necessary to filter and weigh the residue by normal analytical technique.

B-2.4 *Calculation*

The average coating thickness in micrometres is given by the formula:

$$\frac{10\,m}{A \times \rho}$$

where

m = the mass of the coating in milligrams;

A = the area of the gold-plated surface of the specimen in cm^2, and

ρ = the density of the coating in g/cm^3.

Note — The density of pure gold is 19·3 g/cm^3. Electroplated gold often has a lower density because of included organic material or co-deposited alloying metal. If the true density of the coating is not known, use the value 19·3 g/cm^3 but the calculated thickness may then be considerably less than the true thickness.

B-3. Microsection Method

B-3.1 The method given in IS : 3203-1982 is considered to have an accuracy of ±10 percent or ±0·8 µm, whichever is greater.

B-4. The Beta-ray Backscatter Method

B-4.1 This method is considered to have an accuracy of ±10 percent for thickness of gold equal to or greater than 0·5 µm. It must be pointed out that this method requires calibration with gold alloy standard of the same composition as the gold alloy covering to be tested.

B-5. The X-ray Fluorescence Method — This method uses emission and absorption X-ray spectrometry for determining the thickness. It is considered to have an accuracy of better than ±10 percent in the range of 0·5 to 7·5 µm. This method normally determines the mass of gold per unit area. Coating thickness may be calculated from the mass per unit area and the density.

B-6. Interferometric Method

B-6.1 The thickness of the coating is measured by directing a monochromatic light beam upon a step of the gold alloy covering. On a given test sample, this step is produced by selective masking of the gold alloy covering and dissolution of the unmasked area. Dissolution of the gold alloy covering is done preferably by the coulometric method (*see* **B-7.1**). The accuracy of the interferometric method is normally better than 0·2 µm. Since this is a direct method, no calibration is required. This method is used for the calibration of Beta-ray backscatter equipment.

B-7. Coulometric Method

B-7.1 Method mentioned in **B-6.1**, as a preparatory step of the interferometric method, may also be used directly for determining the thickness of gold alloy coverings. In this method, the gold alloy covering is dissolved anodically from a precisely defined area. The thickness is calculated from the quantity of electricity consumed in dissolving the coating from the basis metal. Commercial electrolytes are available for use with gold coatings on copper, nickel and iron alloys. This method is considered to have an accuracy of ±10 percent or ±0·07 µm whichever is greater, for gold coatings less than about 3·5 µm.

APPENDIX C

(*Clause* 5.1)

CORROSION/POROSITY TEST

C-1. General

C-1.1 The various forms of corrosion, which appears on a gold alloy covered article, may be divided into three groups:

a) Corrosion of the basis metal at points where there are gaps in the covering. Electrochemical cells may act at these points and accelerate penetration as also at the boundary between the covering and the basis metal.

b) Attack caused by saline agents or possibly by mildly acidic agents (compact with perspiration, packaging, leathers or certain plastics). The products of corrosion may be of various colours — orange, violet, blue, green or brown.

c) Attack caused by sulphur-containing agents (atmospheric hydrogen sulphide, vulcanized rubber, etc). Such agents may also attack the basis metal at points where there are gaps in the protective covering. They also cause changes in the surface colouring, which may even turn mett black.

C-1.2 The proposed tests make it possible for these various effects to be distinguished to a certain extent. Gold alloy covering shall be resistant in all the environments described below. The development of corrosion is closely allied to the relative humidity of the ambient environment. For this reason, the hygrometric degree shall be determined for each test.

According to the nature of the article, the supplier may, with the agreement of his client; determine the number of items to be submitted to each test and the test conditions. In the test report, the test conditions must be stated.

C-1.3 The tests for determination of the corrosion resistance are applicable to finished items in the condition in which they are supplied to purchasers.

C-1.4 When testing the quality of the coating on significant surfaces, care shall be taken to avoid any usual influence. The sample shall be completely cleaned, first by the use of a water based detergent, then in the mixture of distilled water and ethyl or isopropyl alcohol. Degreasing in a chlorated solvent is insufficient.

C-1.5 The non-significant surfaces of the object shall be coated with lacquer or covering which is sufficiently resistant to prevent any attack on the protected metal throughout the duration of the test.

C-2. Test

C-2.1 Test for a cuperous basis-metal with or without nickel, and die-cast zinc based alloy.

C-2.1.1 A suitable test vessel is a container made of glass or acid resistant plastic capable of being closed by a gas-tight cover or door. Provision shall be made for the support of test specimens within the vessel so that they are about 100 mm above the base. The specimens may be suspended from rods by means of suitable material, for example, nylon thread or glass hooks. All materials used for chamber construction or specimen support shall be non-metallic and inert to the action of acetic acid; glass and an acrylic resin are suitable materials.

C-2.1.2 The test temperature shall be maintained within 23 ± 2°C for the materials under test and the duration of test be 24 hours.

C-2.1.3 During the progress of the test, the test vessel shall be kept screened from draughts or from any source of local radiant heat so that sudden temperature fluctuations and local heating of the test chamber are avoided.

C-2.1.4 The vessel shall be filled with the solution of pure concentrated acetic acid: 25 percent (*m*/*m*) and distilled water 75 percent (*m*/*m*), to a depth of about 10 mm. The walls of the vessel shall be lined with thick white blotting paper which dips into the liquid. Place the test specimens immediately in the vessel, so that they are at least 30 mm from the liquid and the wall of the vessel immediately and move it gently to ensure complete mixing of the two solutions.

C-2.1.5 After expiry of the test period, open the test chamber, remove and inspect the test specimens.

C-2.1.6 *Criteria* — The sample shall not reveal to the naked eye either green droplets or accumulation of green deposits anywhere on the significant surface. On zinc die-cast alloys, no white deposit shall appear.

C-3. Test for a Ferrous Basis-Metal

C-3.1 The test chamber requirements and other test conditions are similar to those described for acetic acid test in **C-2.1**, except that the supersaturated mixture of the following composition shall be used:

a) Crystallized sodium disulphite $Na_2S_2O_5$, 45 percent (*m*/*m*), and

b) Water, 55 percent (*m*/*m*).

C-3.2 *Criteria* — The sample shall not reveal to the naked eye any traces of corrosion anywhere on the significant surface. Slight general tarnishing of low carat coatings is admissible.

C-4. None-Determination of Basis Metal

C-4.1 Where the basic-metal can not be determined, use the test described in **C-2.1** to **C-2.1.6**.

C-5. Testing with Saline and Acid Agents (Synthetic Perspiration Test)

C-5.1 The test shall be carried out in a closed pyrex glass (or equivalent) vessel, which can be heated in an oven to 40°C.

C-5.2 The vessel shall be filled to depth of about 10 mm with the solution having the following composition:

Sodium chloride	20 g/l
Ammonium chloride	17·5 g/l
Urea	5 g/l
Acetic acid	2·5 g/l
Lactic acid	15 g/l
Sodium hydroxide	Quantity required to bring the *p*H to 4·7.

The walls of the vessel shall be lined with thick white blotting paper which dips into the liquid. Place the test specimens immediately in the vessel so that they are at least 30 mm from the surface of the liquid and the wall of the vessel. Then spray a fine mist of the same solution over the surface of the sample using a glass sprayer. Close the vessel immediately and move it gently to ensure complete mixing of the solutions.

C-5.3 Test temperature shall be 40 ± 2°C and the duration of the test 24 hours.

C-5.4 After expiry of the test period, open the test chamber, remove and inspect the test specimens.

C-5.5 *Criteria* — After washing with water, the general colouring of the coating shall not have changed when compared with an untested control sample. The appearance of a light layer of tarnish may be tolerated, provided that it may be removed by wipping the sample. Slight general tarnishing of low carat coatings is admissible.

C-6. Nitric Acid Test

C-6.1 Gold alloy covering shall comply with the test requirements specified in **13** of IS : 3266-1982.

C-7. Thermal Shock Test

C-7.1 Heat the watch case for 5 minutes in an air circulated furnace maintained at 360 to 400°C. Then quench the case in water at an ambient temperature and remove the case from water and dry. Examine the coating for signs of blistering or detachment under magnification of 8X.

EXPLANATORY NOTE

This Indian Standard on gold alloy covering is being issued in the following parts:

Part 1 General requirements

Part 2 Determination of fineness, thickness and corrosion resistance

Part 3 Mechanical properties

This standard is in alignment with ISO 3160/2-1982 'Watch cases and accessories — Gold alloy coverings : Part 2 Determination of fineness, thickness and corrosion resistance', issued by the International Organization for Standardization.

The requirements of thermal shock test and nitric acid test, which are not covered in the above ISO have been included, based on the latest trade practices being followed in the country.

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